



Europäisches Patentamt  
European Patent Office  
Office européen des brevets



(11) Publication number : **0 469 906 A1**

(12)

## EUROPEAN PATENT APPLICATION

(21) Application number : **91307069.4**

(51) Int. Cl.<sup>5</sup> : **C10M 111/04,**  
// (C10M111/04, 101:00,  
103:00, 103:02, 105:24,  
107:02, 107:12, 107:28,  
107:30, 107:38, 107:50)

(22) Date of filing : **01.08.91**

(30) Priority : **02.08.90 JP 204011/90**

(43) Date of publication of application :  
**05.02.92 Bulletin 92/06**

(84) Designated Contracting States :  
**DE GB SE**

(71) Applicant : **OILES CORPORATION**  
**3-2 Shiba-Daimon 1-chome**  
**Minato-ku, Tokyo (JP)**

(72) Inventor : **Chou, Hideo**  
**2-779-44 Zama**  
**Zama-shi, Kanagawa-ken (JP)**  
Inventor : **Sumiyoshi, Kikuo**  
**860-4 Shobuzawa**  
**Fujisawa-shi, Kanagawa-ken (JP)**  
Inventor : **Ishikawa, Keizou**  
**1-9-4 Nishi-Tsuruma**  
**Yamato-shi, Kanagawa-ken (JP)**  
Inventor : **Nishi, Yasunori**  
**273 Ikard Drive**  
**S.E. Concord, N.C. 28025 (JP)**

(74) Representative : **Woods, Geoffrey Corlett et al**  
**J.A. KEMP & CO. 14 South Square Gray's Inn**  
**London WC1R 5LX (GB)**

(54) **Solid lubricant and a sliding member having the solid lubricant embedded therein.**

(57) **A solid lubricant which comprises :**  
5 to 78% by weight of a solid lubricant powder material ;  
5 to 30% by weight of a lubricating oil which is in the form of a liquid or paste at ordinary temperature ;  
2 to 15% by weight of a carrier for absorbing and containing the lubricating oil ; and  
15 to 50% by weight of a synthetic resin binder.

EP 0 469 906 A1

Jouve, 18, rue Saint-Denis, 75001 PARIS

The present invention relates to a solid lubricant having a wettability and a fluidity, and containing a lubricating oil, and a sliding member having the solid lubricant embedded therein.

So-called solid lubricant-embedding sliding members in which apertures or grooves are formed to a metal substrate made of copper alloys or iron alloys and a solid lubricant is embedded and fixed in the apertures or the grooves, have been used for various kind of machine parts. As a manufacturing method for the solid lubricant-embedding sliding member includes, a method① of using a round graphite rod having spiral ridges formed at the outer circumferential surface as a core in a casting mold, casting a molten metal into the casting mold, and removing the core by the cutting, thereby exposing the solid lubricant (graphite) spirally to the sliding surface (Japanese Patent Publication No 39-2506 (1964)); a method② of adhering and fixing solid lubricant pellets (cylindrical form) to paper or film which is burnt to be eliminated upon casting of a molten metal adhering and fixing the obtained solid lubricant pellets to the outer circumferential surface of a core rod composed of a shell sand mold to form a core in the casting mold (Japanese Publication No 56-14381(1981)), or adhering and fixing solid lubricant pellets to a core rod composed of a shell sand mold to form a core for the casting mold (Japanese Patent Publication No 52-5449(1977)), casting a molten metal into the casting mold, thereby exposing the solid lubricant pellets on the sliding surface; or a method③ of forming apertures or grooves to a metal substrate and embedding and fixing solid lubricant pellets coated with adhesives into the apertures or grooves, may be cited.

In the manufacturing methods described above, the method① has drawbacks that the molten metal can not extend sufficiently upon casting since graphite of good heat conductivity is used as it is for the core and the shape of the sliding member is restricted, and the method② has drawbacks that the solid lubricant pellets are detached or displaced during casting and it is difficult to obtain a sliding member in which the solid lubricant pellet are arranged regularly. Accordingly, the method③ described in the above has been used generally.

However, the method③ also involves several technical problems.

(1) Since the size of the apertures (aperture diameter) disposed in the metal substrate is different depending on the size of the metal substrates, the solid lubricant pellets embedded into such apertures having various sizes have to be prepared corresponding to the varying aperture thereof.

(2) Since it is difficult to automate the operation of embedding the solid lubricant pellets coated with adhesives and it has to be down manually, the workability is extremely poor.

(3) Since the solid lubricant pellets can not be used for embedding and fixing solid lubricant into grooves, for example, spiral grooves or ring-shaped grooves disposed in the inner surface of a metal substrate, in particular, a cylindrical metal substrate, it is necessary to provide a paste-like solid lubricant having a fluidity.

Further, as an additional problem, skin eruption or like other unexpected accident may occur to operators during coating operation of adhesives to the solid lubricants in view of (2) described above.

Further, the following solid lubricant have been known.

A lubricant-containing heat-molded composition comprising a polymeric synthetic resinous base material blended with a lubricant adsorbent carrier, the carrier having a lubricant adsorbed thereon, and molded, the carrier having a melting point or a melt viscosity higher than the base material, having a surface area of at least 0.01 m<sup>2</sup>/g, and being selected from the group consisting of

- ① a non-fusible inorganic material;
- ② a synthetic high molecular weight polymeric resinous material;
- ③ a naturally occurring high molecular weight polymeric material; and
- ④ mixtures thereof;

and the base material being selected from the group consisting of a polymeric synthetic resin and a mixture of at least two polymeric synthetic resins (U.S. Patent No. 3778918).

A sliding surface embedding-type solid lubricant comprising a solid lubricant powder, a thermosetting synthetic resin or an ordinary temperature setting-type synthetic resin which is liquid under an ordinary temperature for bonding the powdery particles and a lubricating oil impregnated between each of the powdery particles bonded by the synthetic resin (Japanese Patent Application Laid-Open (KOKAI) No. 58-160398(1983)).

A composite lubricating material which can be molded into a desired shape and have a high surface strength, comprising 0.5 to 90.0 % by weight of a pulverized, surface-treated and expanded graphite product (a + b) obtained by sujeacting from 5 to 99.5 % by weight of an expanded graphite powder (a) having an apparent specific gravity of 0.01 to 0.50 obtained by wet oxydation of natural graphite, kish graphite and artificial graphite, and then expanding and disintegrating them under a high temperature heat treatment, to surface treatment with 0.5 to 95.0 % by weight of a lubricating oil (b), for example, turbine oil, machine oil, spindle oil and bearing oil, and 10 to 99.5 % by weight of an organic binder (c) composed of a thermosetting resin such as phenol resin, melamine resin, epoxy resin and polyimide resin, or a thermoplastic resin such as polyphenylene sulfide, polyacetal, polytetrafluoroethylene, nylon 6, nylon 11 and polyurethane (Japanese Patent Application Laid-

Open (KOKAI) No. 63-23993(1988)).

An extrudable or injection moldable self-lubricating composition comprising 100 parts by weight of a molding product composed of 99 to 10 parts by weight of a lubricating oil and 20 to 90 parts by weight of a super high molecular weight polyethylene, and 0.1 to 20 parts by weight of a powder blended therewith (Japanese Patent Application Laid-Open (KOKAI) No. 63-161075(1988)).

On the other hand, under the consideration of the performance of the sliding member, since the embedded solid lubricant is supplied little by little to the sliding surface to form a solid lubricant film, it can be served for the long time use without additionally supplying the lubricant. However, since the application use is substantially restricted to that in a low speed and high load region, the combined use with a lubricating oil is inevitable in an application use as being out of the above-mentioned region.

Accordingly, in the solid lubricant-embedding sliding member to be used in the application use out of the above-mentioned region, it is required to form grooves to the sliding surface as grease pits or to supply the lubricating oil from the oil feeding device to the sliding surface.

For the foregoing problems in view of manufacture and performance, the present inventors have considered that the all of the problems described above can be overcome thoroughly by satisfying all of the technical subjects, namely, ① solid lubricant can be embedded without restrictions at all, to the size and the shape of the apertures or grooves disposed in the metal substrate, ② the solid lubricant can provide both of the lubricating effect by the lubricating oil and the lubricating effect by the solid lubricant, and ③ the solid lubricant can be bonded to the apertures or grooves in the metal substrate and, as a result of an earnest study, it has been found that by blending a solid lubricant powder material with a lubricating oil which is in a liquid or paste-like form under an ordinary temperature, a carrier for absorbing and possessing the lubricating oil, and a synthetic resin binder, the thus-obtained solid lubricant has a wettability and a fluidity and is bonded to the apertures or grooves disposed in the metal substrate even if it contains the lubricating oil. On the basis of the finding, the present invention has been attained.

As a first aspect of the present invention, there is provided a solid lubricant having a wettability and a fluidity, comprising 5 to 78 % by weight of a solid lubricant powder material, 5 to 30% by weight of a lubricating oil which is in a liquid or paste form at an ordinary temperature, 2 to 15% by weight of a carrier for absorbing and possessing the lubricating oil, and 15 to 50% by weight of a synthetic resin binder.

In a second aspect of the present invention, there is provided a sliding member having a solid lubricant embedded therein in which the solid lubricant defined in the first aspect is charged into apertures or grooves disposed in a metal substrate and said solid lubricant is bonded to the apertures or grooves in the metal substrate.

Fig. 1 is a vertical cross sectional view of one embodiment of a flow charging device for a solid lubricant;

Fig. 2 is a cross sectional view taken along line II-II in Fig. 1; and

Fig. 3 is a vertical cross sectional view illustrating a sliding member having a solid-lubricant embedded therein.

In the present invention, the solid lubricant powder material as the main ingredient is a material having a solid lubricating effect by itself. For example, one or more of materials selected from the group consisting of: (1) natural graphite, (2) artificial graphite obtained by molding an aggregate such as a coke powder using a binder such as coal tar pitch which is carbonized and graphitized by itself under heating and burning, baking the maled product a temperature of 700 to 1100°C, permeating an impregnating agent such as pitch, and heat-treating at a temperature of 2500 to 3000°C to graphitize the permeated product, (3) an expanded graphite prepared by treating natural flaky graphite, kish graphite, thermally decomposable graphite or the like with a strong acid to form a inter-graphite layer compound followed by washing with water and then subjecting to an expanding treatment in the direction of the C-axis instantaneously at high temperature, (4) molybdenum disulfide, (5) polytetrafluoroethylene resin (PTFE) and (6) boron nitride may be exemplified.

A solid lubricant powder material with an average grain size of not more than 150 µm, preferably 80 to 130 µm is preferred.

The blending amount of the solid lubricant power material is determined as the balance of the lubricating oil, the carrier and the synthetic resin binder to be blended, and it is within a range from 5 to 78 % by weight, preferably from 33 to 74 % by weight, more preferably 47 to 67 % by weight.

As the lubricating oil which is liquid or paste under an ordinary temperature, mineral oils such as machine oil and engine oil, vegetable oils such as castor oil, synthetic oils such as ester oil and silicone oil, and grease may be exemplified, and one or more them is used.

When the lubricating oil is mixed with the solid lubricant powder material described above and the carrier for absorbing and possessing the lubricating oil, most of the lubricating oil is absorbed and possessed in the carrier and a portion thereof is adsorbed and retained, at the surface of the solid lubricant powder material.

The blending amount of the lubricating oil is from 5 to 30 % by weight, preferably from 8 to 20 % by weight,

more preferably 10 to 15 % by weight. If it is blended in excess of 30 % by weight, the adsorption amount of the lubricating oil to the surface of the solid lubricant powder material is increased and the lubricant oil is present at the bonding boundary between the solid lubricant and the apertures or the grooves in the metal substrate, to weaken the bonding strength of the solid lubricant with the apertures or grooves in the metal substrate, thereby bringing about a defect in which the solid lubricant is drawn out of from the apertures or the grooves in the metal substrate. Further, if the blending amount is less than 5% by weight, although the bonding strength of the solid lubricant to the apertures or the grooves in the metal substrate is increased, the bleeding amount of the lubricating oil to the sliding surface is reduced, thereby failing to obtain an improvement for the sliding performance by the lubricating oil.

10 The carrier is blended together with the solid lubricant powder material and the lubricating oil described above, and it has a characteristic of an absorbing and possessing the lubricating oil.

The carrier has a function of absorbing and possessing the lubricating oil, thereby increasing the flow viscosity of the lubricating oil, preventing to bleed out the lubricating oil, and supplying the lubricating oil which is uniformly dispersed and absorbed and possessed in the solid lubricant, to the sliding surface.

15 As the carrier usable in the present invention, (1) a hydrocarbon wax, (2) a higher fatty acid, (3) a waxy material such as wax derived from the higher fatty acid, (4) a polyolefin powder, (5) an oleophilic fiber, (6) crosslinked porous spherical particles mainly composed of styrene or methacrylic material, and (7) a porous calcium carbonate, may be exemplified. They may be used alone or as a mixture of two or more of them.

20 (1) Hydrocarbon Wax

The hydrocarbon wax can include a paraffin wax having not less than 24 of carbon atoms, an olefin wax having not less than 26 of carbon atoms and an alkylbenzene having not less than 28 of carbon atoms, and in addition, a crystalline microcrystalline wax can also be used.

25 (2) Higher Fatty Acid

As the higher fatty acid, there can be mentioned a saturated fatty acid having not less than 14 of carbon atoms, for example, myristic acid, palmitic acid, stearic acid, arachic acid and montanic acid, as well as an unsaturated fatty acid having not less than 18 of carbon atoms, for example, octadecenoic acid and parinaric acid.

(3) Waxy Material Derived from Higher Fatty Acid

35 As the waxy material, there can be mentioned, for example, (a) a higher fatty acid ester, (b) a higher fatty acid amide and (c) a higher fatty acid salt.

(a) Higher fatty acid ester

40 As the higher fatty acid ester, there can be mentioned a methyl and ethyl ester of higher fatty acid having not less than 22 of carbon atoms, for example, ethyl behenate and methyl tricosane; an ester of a mono-valent higher fatty acid having not less than 16 of carbon atoms and a higher monohydric alcohol having not less than 15 of carbon atoms; octadecyl stearate, a monodiglycerine ester of higher fatty acid having not less than 13 of carbon atoms, for example, 1,3-dilauric acid glyceride, monostearic acid glyceride; and a triglyceride of higher fatty acid having not less than 14 of carbon atoms.

(b) Higher fatty acid amide

45 As the higher fatty acid amide, there can be mentioned, in particular, palmitic acid amide, stearic acid amide and oleic acid amide.

(c) Higher fatty acid salt

50 As the higher fatty acid salt, there can be mentioned salt with alkali and alkaline earth metal, for example, lithium stearate and calcium stearate.

## (4) Polyolefin

As the polyolefin, there can be mentioned a super higher molecular weight polyethylene powder, a high, medium and low density polyethylene powder and a polypropylene powder.

5

## (5) Oleophilic Fiber

As the oleophilic fiber, there can be mentioned a cellulose fiber and a polypropylene fiber.

## 10 (6) Crosslinked porous spherical particles mainly composed of styrene or methacrylic material

As the porous spherical particles, there can be mentioned, for example, styrene-divinylbenzene copolymer.

## 15 (7) Porous Calcium Carbonate

The blending amount of the carrier is generally from 2 to 15 % by weight, preferably 3 to 12 % by weight, more preferably 3 to 8 % by weight.

A most preferred combination can be obtained for the bonding strength of the solid lubricant to the apertures or the grooves in the metal substrate and the lubricating property by setting the amount of the carrier toward the lower limit when the lubricating oil to be blended is on the side of the lower limit, whereas by setting the blending amount of the carrier toward the upper limit when the blending amount of the lubricating oil is on the side of the upper limit.

The synthetic resin binder has roles of bonding the solid lubricant powder materials to each other and of bonding the solid lubricant with the apertures or the grooves in the metal substrate. As the synthetic resin binder, there can be mentioned a thermosetting synthetic resin such as epoxy resin, phenol resin and polyester resin, and the epoxy resin is preferred in the present invention.

As the epoxy resin, an ordinary temperature setting liquid epoxy resin and a thermosetting liquid or powdery epoxy resin are used.

Specifically, as the ordinary temperature setting epoxy resin, there can be mentioned a two component epoxy resin of "Cemedyne" (trade name, produced by Cemedyne Co.)

Further, as the thermosetting liquid epoxy resin, there can be mentioned a one component epoxy resin of "Technodyne" (trade name, produced by Taoka Kagaku Co.), a liquid epoxy resin "Epicoat" (trade name, produced by Yuka Shell Epoxy Co.), a latent curing agent "Novacure" (trade name, produced by Asaki Kasei Industry Co.) and "Amicure" (trade name, produced by Ajinomoto Co.)

As the thermosetting powdery epoxy resin, there can be mentioned "Epiform" (trade name, manufactured by Somal Co.), and the powdery epoxy resin may also be used as a flow controlling agent for the liquid epoxy resin.

The blending amount of the binder is from 15 to 50 % by weight, preferably 15 to 35 % by weight, more preferably 20 to 30 % by weight.

40 The solid lubricant in the present invention is prepared as shown below.

A carrier absorbing and possessing the lubricating oil is blended and mixed with a solid lubricant powder material and then the lubricating oil is blended and mixed with the thus-obtained mixture. Then, a synthetic resin binder is added to the resultant mixture of the solid lubricant powder material, the carrier and the lubricating oil, and they are kneaded to obtain a solid lubricant having a wettability and a fluidity.

45 The thus obtained solid lubricant having a wettability and a fluidity is flow-charged at an ordinary temperature or under the heating into the apertures or the grooves disposed in the metal substrate, and subsequently, the synthetic resin binder therein is cured at an ordinary temperature or under the heating to bond the solid lubricant containing the lubricating oil with the apertures or the grooves in the metal substrate, thereby obtaining a sliding member embedded with a solid lubricant containing the lubricating oil. As a method of flow-charging the solid lubricant having a wettability and a fluidity into the apertures or the grooves disposed in the metal substrate, transfer molding, compression molding, injection molding or the like may be used. In case of a plate-like sliding member, there can be used a method of previously molding the solid lubricant into a plate-like shape, placing the thus-obtained plate-like solid lubricant on a plate-like sliding part formed with apertures or grooves and then flow charging the solid lubricant by press molding.

55 Fig. 1 is a cross sectional view illustrating one embodiment of a flow-charging device for the solid lubricant and Fig. 2 is a cross sectional view taken along line II-II in Fig. 1.

In the drawing, there are illustrated a holder 1 having a cylindrical portion 11; a sliding member material (metal substrate) 2 fitted into the cylindrical portion 11 of the holder 1 with its outer circumferential surface to

be constrained, and having apertures 21 formed such that they are overlapped to each other in the outer circumferential direction; a guide member 3 having a radial flow channel 31 formed at the upper end surface and inserted to the inner surface of the sliding member material 2 with a slight gap S relative to the inner diametrical surface of the material 2; a vessel 4 fitted to the upper end of the cylindrical portion 11 of the holder 1 which holds the material 2 and having a flow channel 41 in communication with the flow channel 31; a solid lubricant 5 having a wettability and a fluidity charged in the vessel 4; and a pressing tool 6 fitted vertically movably in the vessel 4.

Fig. 3 is a cross sectional view illustrating a solid lubricant embedding sliding member manufactured by using the apparatus as described above, in which are shown a sliding member 7, apertures 71 formed in the sliding member material and solid lubricant 72 embedded in the apertures 71.

The bonding strength between the solid lubricant and the metal substrate in the sliding member having the solid lubricant embedded therein according to the present invention is not less than 50 kgf/cm<sup>2</sup>. At a 200 mm stroke of a reciprocating sliding test and after 100 x 10<sup>4</sup> test cycles (400 Km test stroke), by using the mechanical structural carbon steel (S45C) as the mating member, ① a friction coefficient is not more than 0.135 and the abrasion amount is not more than 0.0125 mm under a load of 500 kgf/cm<sup>2</sup> and at a sliding speed at 1 m/min; and ② a friction coefficient is not more than 0.125 and abrasion amount is not more than 0.0105 mm under a load at 10 kgf/cm<sup>2</sup> and at a sliding speed at 40 m/min.

The solid lubricant and the sliding member according to the present invention have effects as described below.

- 20 (1) Since the solid lubricant has a wettability and a fluidity, it can be embedded without restriction at all of the shape of the apertures or grooves disposed in the metal substrate.
- 25 (2) Since the solid lubricant contains in itself a lubricating oil, the lubricating oil possessed by the carrier in the solid lubricant is supplied to the sliding surface during sliding movement relative to the mating member by which lubricating effect of the solid lubricant and the lubricating oil can be conducted. Accordingly, it is no more necessary to supply additional lubricant such as grease to the sliding surface as in the prior art for the solid lubricant-embedding sliding member under a low speed and high load condition, as well as for the application use out of such conditions, and the use under wide range of conditions is enabled.
- 30 (3) Since the lubricating oil is absorbed and possessed in the carrier, the bleeding out thereof is extremely small and a great amount of the lubricating oil is contained in the solid lubricant.
- 35 (4) Since the solid lubricant is embedded by flow charging into the apertures or the grooves disposed in the metal substrate, conventional adhesive coating operation or embedding operation in the prior art is no more necessary to remarkably improve the operability, as well as troubles such as skin eruption of operators caused by the adhesive coating operation can be overcome thoroughly.

#### 35 <Example>

The solid lubricant and the sliding member having the solid lubricant embedded therein according to the present invention will be explained more in details referring to examples.

#### 40 Example I

- 45 (1) 53 % by weight of an artificial graphite powder obtained by pulverizing an artificial porous graphite molding product having an apparent specific gravity of 1.55 g/cm<sup>3</sup> and a porosity of 45 % (manufactured by Ibigden Co.), and preparing to an average grain size of 100 µm.
- 50 (2) 43 % by weight of the artificial graphite powder described in (1) above and 10% by weight of a polytetrafluoroethylene (PTFE) powder ("Yunon" (trade name), manufactured by Nippon Vulker Co.), and (3) 43 % by weight of the artificial graphite powder as described in (1) above and 10% by weight of a molybdenum disulfide powder, were used as the solid lubricant powder material; 5 % by weight of a hydrocarbon wax ("Godeswax" (trade name), manufactured by Nikko Fine Products Co.) as a carrier were added with the solid lubricant powder material by a mixer; and 15 % weight of a mineral oil as a lubricating oil ("Daffny Super Multi" (trade name), manufactured by Idemitsu Kosan Co.) were mixed with the resultant mixture to obtain a mixture of the solid lubricant powder material, the carrier and the lubricating oil.
- 55 Then, 16 % by weight of a thermosetting liquid epoxy resin ("Epicoat" (trade name), manufactured by Showa Shell Chemical Co.) as a synthetic resin binder, 5 % by weight of a latent-type setting agent ("Novacure" (trade name), manufactured by Asahi Kasei Industry Co.) and 6 % by weight of a powdery epoxy resin ("Epiform" (trade name), manufactured by Somal Co.) were mixed with the thus-obtained mixture for flow control and then kneaded to obtain a solid lubricant having a wettability and a fluidity.

A high strength brass casting product (JIS-H2205) was used as the metal substrate, which was fabricated by cutting into a cylindrical substrate of 60 mm inner diameter, 80 mm outer diameter and 80 mm length, and 54 apertures each of 10 mm diameter were formed to the cylindrical substrate so as to overlap to each other in the circumferential direction of the cylindrical substrate to form a sliding member material.

5 While constraining the sliding member material at the outer circumferential surface, the solid lubricant was caused to flow at an ordinary temperature under a pressure of 50 kg/cm<sup>2</sup> from the inner circumferential surface of the material by the flow discharging device shown in Fig. 1 and was tightly charged into the apertures of the material.

10 Subsequently, the sliding member material having the solid lubricant held in the apertures was kept in a heating furnace to a temperature of 80°C for 60 min to conduct primary setting for the synthetic resin binder and then further kept in the heating furnace to a temperature of 140°C for 30 min to conduct the secondary setting for the synthetic resin binder. At the same time, the solid lubricant containing the lubricating oil was bonded to the apertures of the material, thereby obtaining the sliding member having the solid lubricant embedded therein (bearing bush).

15 Example II

(1) 40 % by weight of an expanded graphite powder obtained by pulverizing an expanded graphite sheet having an apparent specific gravity of 0.7 g/cm<sup>3</sup> and a thickness of 0.5 mm ("Nikafilm" (trade name), manufactured by Nippon Carbon Co.), and preparing to an average grain size of 120 µm.

20 (2) 20 % by weight of the expanded graphite powder, and 20 % by weight of the artificial graphite powder, and

25 (3) 20 % by weight of the expanded graphite powder and 20 % by weight of a boron nitride powder, were used as the solid lubricant powder material; 8 % by weight of a higher fatty acid (stearic acid) was added as a carrier to the solid lubricant powder material and mixed by a mixer; and 17 % by weight of a mineral oil as a lubricating oil (same as in Example I) were mixed with resultant mixture to obtain a mixture of the solid lubricant powder material, the support or the carrier and the lubricating oil.

30 Then, 21 % by weight of a thermosetting liquid epoxy resin (same as in Example I) as a synthetic resin binder, 6 % by weight of a latent type curing or setting agent (same as in Example I) and 8 % by weight of a powdery epoxy resin (same as in Example I) for flow control were mixed with the thus-obtained mixture and then kneaded to obtain a solid lubricant having a wettability and a fluidity.

35 After manufacturing a sliding member material by using the same high strength brass casting product as in Example I as the metal substrate, the solid lubricant was tightly charged in the apertures of the material in the same procedures as those in Example I.

Subsequently, the solid lubricant-embedding sliding member was obtained in the procedures as those in Example I.

Example III

40 (1) 35 % by weight of the same artificial graphite powder as in Example I,

(2) 20 % by weight of the artificial graphite powder and 15 % by weight of a polytetrafluoroethylene powder (as in Example I), and

45 (3) 20 % by weight of the artificial graphite powder and 15 % by weight of a molybdenum disulfide powder, were used as the solid lubricant powder material; 7 % by weight of a super high molecular weight polyethylene powder ("Hizexmillion" (trade name), manufactured by Mitsui Petrochemical Industry Co.), 3% by weight of a higher fatty acid amide (mixture of oleic acid and stearic acid) were added as a carrier to the solid lubricant powder material and mixed by a mixer; and 20 % by weight of a mineral oil as a lubricating oil (same as in Example I) were mixed with the resultant mixture to obtain a mixture of the solid lubricant powder material, the support or the carrier and the lubricating oil.

50 Then, 21 % by weight of a thermosetting liquid epoxy resin (same as in Example I) as the synthetic resin binder, 6 % by weight of a latent type setting agent (same as in Example I) and 8 % by weight of a powdery epoxy resin (same as in Example I) for flow control were mixed with the thus-obtained mixture and then kneaded to obtain a solid lubricant having a wettability and a fluidity.

55 After manufacturing a sliding member material by using the same high strength brass casting product as in Example I as the metal substrate, the solid lubricant was tightly charged in the apertures of the material in the same procedures as those in Example I.

Subsequently, the solid lubricant-embedding sliding member was obtained in the same procedures as those in Example I.

Example IV

5 (1) 70 % by weight of a molybdenum disulfide powder,  
 (2) 60 % by weight of the molybdenum disulfide powder and 10 % by weight of a polytetrafluoroethylene powder (as in Example I), were used as the solid lubricant powder material; 5 % by weight of a hydrocarbon wax (the same as in Example I) was added as a carrier to the solid lubricant powder material and mixed by a mixer, and 8 % by weight of mineral oil as a lubricating oil (same as in Example I) were mixed with the thus-obtained mixture to obtain a mixture of the solid lubricant powder material, the support or the carrier and the lubricating oil.

10 Then, 11 % by weight of a thermosetting liquid epoxy resin (same as in Example I) as a synthetic resin binder, 3 % by weight of a latent type setting agent (same as in Example I) and 4 % by weight of a powdery epoxy resin (same as in Example I) for flow control were mixed with the thus-obtained mixture and then kneaded to obtain a solid lubricant having a wettability and a fluidity.

15 After manufacturing a sliding member material by using the same high strength brass casting product as in Example I as the metal substrate, the solid lubricant was tightly charged in the apertures of the material in the same procedures as those in Example I.

Subsequently, the solid lubricant-embedding sliding member was obtained in the same procedures as those in Example I.

Example V

20 (1) 53 % by weight of the same artificial graphite powder as in Example I,  
 (2) 43 % by weight of the artificial graphite powder and 10 % by weight of a polytetrafluoroethylene (PTFE) powder (same as in Example I), and  
 (3) 43 % by weight of the artificial graphite powder and 10 % by weight of a boron nitride powder were used as the solid lubricant powder material; 5 % by weight of a porous styrene-divinylbenzene copolymer powder ("Techpolymer" (trade name) manufactured by Sekisui Kaseihin Kogyo Co.) as a carrier were added with the solid lubricant powder material by a mixer, and 15 % by weight of a mineral oil as a lubricating oil (same as in Example I) were mixed with the resultant mixture to obtain a mixture of the solid lubricant powder material, the support or the carrier and the lubricating oil.

25 Then, 16 % by weight of a thermosetting liquid epoxy resin (same as in Example I) as a synthetic resin binder, 5 % by weight of a latent type setting agent (same as in Example I) and 6 % by weight of powdery epoxy resin (same as in Example I) for flow control were mixed with the resultant mixture and then kneaded to obtain a solid lubricant having a wettability and a fluidity.

30 After manufacturing a sliding member material by using the same high strength brass casting product as in Example I as the metal substrate, the solid lubricant was tightly charged in the apertures of the material in the same procedures as those in Example I.

35 Subsequently, the solid lubricant-embedding sliding member was obtained in the same procedures as those in Example I.

40 Then, the results of tests for the bonding strength of the solid lubricant and the sliding characteristics of the sliding member regarding the solid lubricant and the sliding member having the solid lubricant embedded therein obtained in the examples described above were set forth below.

## (Bonding Strength Test)

45 For the bonding strength, a push-pull gauge was placed on the solid lubricant embedded into the apertures of the sliding member to measure the drawing-out force (kgf/cm<sup>2</sup>) of the solid lubricant from the apertures and the bonding strength was expressed by the measured value.

## (Sliding Property Test)

50 For the sliding property, a friction coefficient and an abrasion amount were measured under the following test conditions.

### (Test Conditions)

(1)	Load:	500 kgf/cm <sup>2</sup>
	Speed:	1 m/min
	Mating member:	Carbon steel for machine structure use (S45C)
	Reciprocating	
	Sliding test:	Stroke 200 mm
	Test cycle:	100 x 10 <sup>4</sup> (400 km)
(2)	Load:	10 kgf/cm <sup>2</sup>
	Speed:	40 m/min
	Mating member:	Carbon steel for machine structure use (S45C)
	Reciprocating	
	Sliding test:	Stroke 200 mm
	Test cycle:	10 x 10 <sup>4</sup> (400 km)

The test results are shown in the following table.

35

40

45

50

55

5  
10  
15  
20  
25  
30  
35  
40  
45  
50  
55

Table

	Solid lubricant ingredient composition (wt%)			Test condition (1)			Test condition (2)	
	Solid lubricant powder material	Carrier	Lubricating oil	Resin binder	Bonding strength (Kgf/cm <sup>2</sup> )	Friction coefficient	Abrasion amount (mm)	Friction coefficient
Example I	① Artificial graphite: 53	Hydrocarbon wax: 5	Mineral oil: 15	27	62	0.09~0.10	0.008	0.07~0.09
	② Artificial graphite: 43 PTFE: 10				58	0.09~0.11	0.010	0.09~0.10
	③ Artificial graphite: 43 MoS <sub>2</sub> : 10				60	0.10~0.11	0.012	0.09~0.11
Example II	① Expanded graphite: 40	Higher fatty acid: 8	Mineral oil: 15	35	50	0.10~0.12	0.009	0.09~0.10
	② Expanded graphite: 20 PTFE: 20				60	0.10~0.11	0.008	0.09~0.11
	③ Expanded graphite: 20 BN: 20				68	0.11~0.13	0.011	0.10~0.12
Example III	① Artificial graphite: 35	Super high molecular weight polyethylene: 7	Mineral oil: 20	35	66	0.08~0.10	0.007	0.07~0.09
	② Artificial graphite: 20 PTFE: 15	Higher fatty acid amide: 3			65	0.08~0.11	0.008	0.08~0.10
	③ Artificial graphite: 20 MoS <sub>2</sub> : 15				58	0.09~0.11	0.010	0.08~0.10
Example IV	① MoS <sub>2</sub> : 70	Hydrocarbon wax: 5	Mineral oil: 7	18	65	0.10~0.12	0.011	0.09~0.11
	② MoS <sub>2</sub> : 60 PTFE: 10				60	0.10~0.11	0.010	0.09~0.10
Example V	① Artificial graphite: 63	Porous styrene-divinyl benzene copolymer: 5	Mineral oil: 15	27	60	0.09~0.10	0.008	0.07~0.09
	② Artificial graphite: 43 PTFE: 10				56	0.09~0.11	0.010	0.09~0.10
	③ Artificial graphite: 43 MoS <sub>2</sub> : 10				58	0.09~0.11	0.012	0.09~0.11

Table (continued)

Comp. Example		Solid lubricant ingredient composition (wt%)	Bonding strength (Kgf/cm <sup>2</sup> )	Test condition (1)		Test condition (2)	
				Friction coefficient	Abrasion amount (mm)	Friction coefficient	Abrasion amount (mm)
①	Graphite pellets bonded to apertures with adhesives (non lubrication)		70	0.12~0.30	0.045	—*	—*
②	Graphite pellets bonded to apertures with adhesives (gerase coated on the sliding surface)		70	0.09~0.13	0.013	0.09~0.35	0.035**

(Note) In the Table, the abrasion amount (mm) in Example V was measured as the change of size for the sliding surface of the sliding member after elapse of the test stroke (400 km).

\* : Since abrupt increase was observed for the friction coefficient and the abrasion amount at the 1 km test stroke the subsequent test was interrupted.

\*\* : Measured value for the abrasion amount at 300 km test stroke..

From the results of the test, the bonding strength of the solid lubricant of the examples to the apertures in the sliding member (drawing-out force) showed lower values than those in the prior art shown in comparative examples.

However, the solid lubricant containing the lubricating oil in the Examples was bonded with a bonding strength of not less than 50 kgf/cm<sup>2</sup>, sliding properties in the low speed/high load region (under test conditions (1) above) in the appropriate application use for the solid lubricant-embedding sliding member, as well as in other application uses out of the low speed/high load region (under test conditions (2) above) were superior to those of the prior art, and the solid lubricant in the Examples caused no troubles at all in using the sliding members.

In the sliding member of Comparative Example 1, the friction coefficient was gradually increased with lapse of test time in the test under the test condition (1) and the abrasion amount was abruptly increased at 300 km test stroke. Further, since the bearing temperature, friction coefficient and abrasion amount were increased abruptly at 1 km of the test stroke after the start of the test under the test conditions (2) above, the test was interrupted.

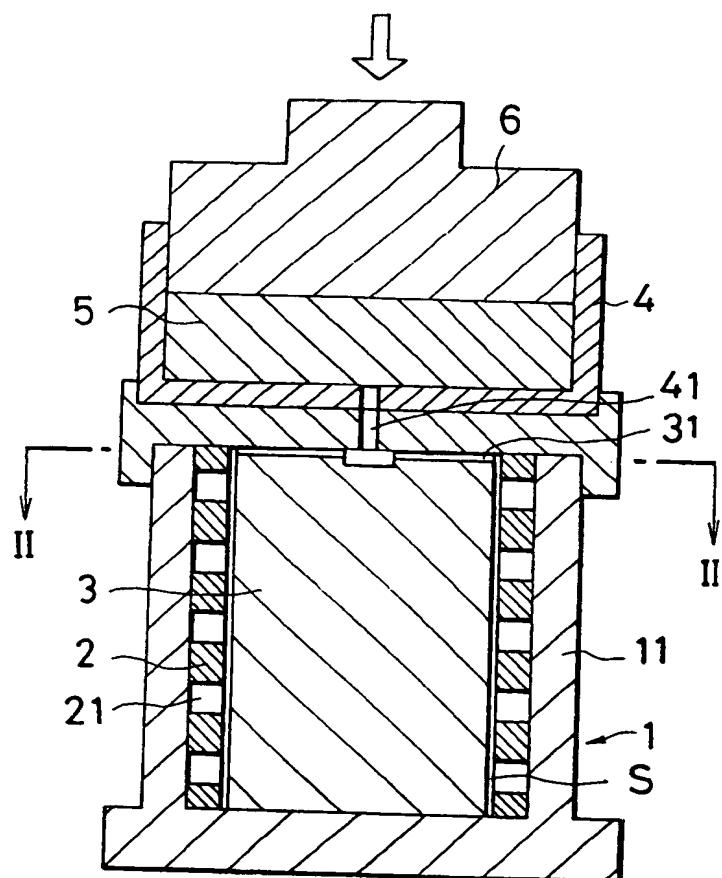
The sliding member of Comparative Example (2) showed substantially the same performance as the sliding member of Example I through Example V in the test under the test conditions (1), but grease coated on the sliding surface began to be squeezed out to the sliding surface at the test stroke of 200 km and both of the friction coefficient and the abrasion amount increased abruptly at the test stroke of 300 km in the test under the test conditions (2) and, accordingly, subsequent test was interrupted.

On the other hand, since the lubricating oil is contained in the solid lubricant of the Examples, it exhibited stable performance without additionally supplying a lubricant not only in the low speed/high load region (the test condition (1)) as the appropriate application use for the solid lubricant-embedding sliding member but also in other application uses out of the such region (the test condition (2)).

## 25 Claims

1. A solid lubricant which comprises:
  - 5 to 78% by weight of a solid lubricant powder material;
  - 30 5 to 30% by weight of a lubricating oil which is in the form of a liquid or paste at ordinary temperature;
  - 2 to 15% by weight of a carrier for absorbing and containing the lubricating oil; and
  - 15 to 50% by weight of a synthetic resin binder.
2. A lubricant according to claim 1 wherein the solid lubricant powder material is at least one of graphite, molybdenum disulfide, a polytetrafluoroethylene resin or boron nitride.
3. A lubricant according to claim 1 or 2 wherein the lubricating oil is at least one of a mineral oil, vegetable oil, synthetic oil or grease.
- 40 4. A lubricant according to claim 3 wherein the lubricating oil is at least one of a machine oil, engine oil, castor oil, ester oil or silicone oil.
5. A lubricant according to any one of the preceding claims wherein the carrier is at least one of a hydrocarbon wax, higher fatty acid, wax derived from a higher fatty acid, polyolefin, oleophilic fibers, crosslinked porous 45 spherical particles composed of a styrene or methacrylic compound or porous calcium carbonate.
6. A lubricant according to any one of the preceding claims wherein the synthetic resin binder is a thermosetting synthetic resin.
- 50 7. A sliding member comprising a lubricant as defined in any one of the preceding claims embedded in apertures or grooves disposed in a metal substrate and bonded thereto.
8. A member according to claim 7 wherein the sliding surface of the metal substrate is a flat, cylindrical or spherical surface.
- 55 9. A process for producing a member as defined in claim 7 or 8, which comprises flowing the lubricant into the apertures or grooves of the substrate.

*Fig. 1*



*Fig. 2*

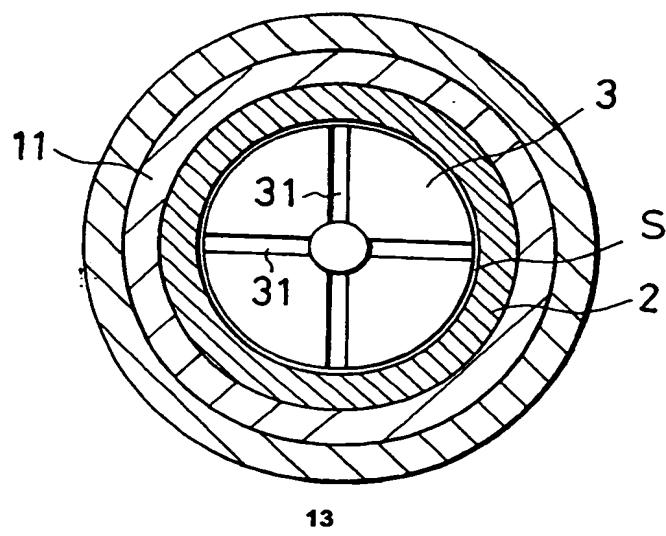
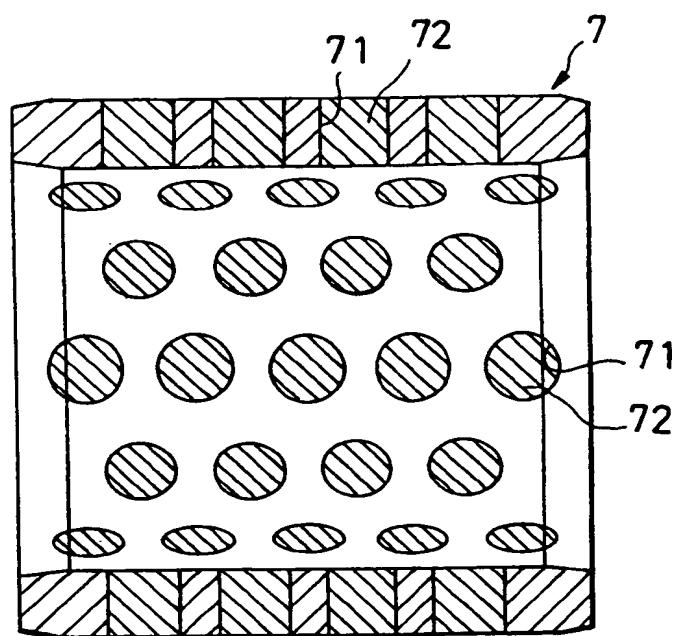


Fig. 3





European Patent  
Office

## EUROPEAN SEARCH REPORT

Application Number

EP 91 30 7069

DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. 5)												
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim													
Y	EP-A-0 116 882 (LONZA) * Whole document *	1-9	C 10 M 111/04 // (C 10 M 111/04												
Y	GB-A-2 223 504 (DURAFILM) * Whole document *	1-5	C 10 M 101:00 C 10 M 103:00 C 10 M 103:02												
D, Y	DERWENT, WORLD PATENT INDEX LATEST, AN=83-804423, Derwent Publications Ltd, London, GB; & JP-A-58 160 398 (MISUMI SHOJI K.K.) ---	1-9	C 10 M 105:24 C 10 M 107:02 C 10 M 107:12 C 10 M 107:28 C 10 M 107:30												
Y	EP-A-0 372 559 (TAKATA CORP.) * Whole document *	1-6	C 10 M 107:38 C 10 M 107:50 )												
Y	US-A-4 486 319 (JAMISON) * Whole document *	1-6													
A	EP-A-0 000 697 (SKF IND. INC.) * Whole document *	1-9													
			TECHNICAL FIELDS SEARCHED (Int. Cl. 5)												
			C 10 M												
<p>The present search report has been drawn up for all claims</p> <table border="1" style="width: 100%; border-collapse: collapse;"> <tr> <td style="width: 33%;">Place of search</td> <td style="width: 33%;">Date of completion of the search</td> <td style="width: 34%;">Examiner</td> </tr> <tr> <td>THE HAGUE</td> <td>08-11-1991</td> <td>DE LA MORINERIE B.M.S.E.</td> </tr> <tr> <td colspan="2">CATEGORY OF CITED DOCUMENTS</td> <td></td> </tr> <tr> <td colspan="2">           X : particularly relevant if taken alone            Y : particularly relevant if combined with another document of the same category            A : technological background            O : non-written disclosure            P : intermediate document         </td> <td>           T : theory or principle underlying the invention            E : earlier patent document, but published on, or after the filing date            D : document cited in the application            L : document cited for other reasons            A : member of the same patent family, corresponding document         </td> </tr> </table>				Place of search	Date of completion of the search	Examiner	THE HAGUE	08-11-1991	DE LA MORINERIE B.M.S.E.	CATEGORY OF CITED DOCUMENTS			X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons A : member of the same patent family, corresponding document
Place of search	Date of completion of the search	Examiner													
THE HAGUE	08-11-1991	DE LA MORINERIE B.M.S.E.													
CATEGORY OF CITED DOCUMENTS															
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons A : member of the same patent family, corresponding document													